SYNTHESIS AND CHARACTERIZATION OF THERMORESPONSIVE POLY (N-VINYLCAPROLACTAM) / FILLERS NANOCOMPOSITE

MARWAH NOORI MOHAMMED

Doctor of Philosophy

UNIVERSITI MALAYSIA PAHANG

SUPERVISOR'S DECLARATION

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Doctor of Philosophy.

_____

(Supervisor's Signature)

Full Name : DR. KAMAL BIN YUSOH

Position : ASSOCIATE PROFESSOR

Date :

_____

(Co-supervisor's Signature)

Full Name : DR. JUN HASLINDA BINTI HAJI SHARIFFUDDIN

Position : SENIOR LECTURER

Date :

STUDENT'S DECLARATION

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

_____

(Student's Signature)

Full Name : MARWAH NOORI MOHAMMED

ID Number : PKC15019

Date : APRIL 2019

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ABSTRAK

Poly (N-vinylcaprolactam) (PNVCL) telah menarik perhatian banyak penyelidikan baru-baru ini sebagai salah satu polimer thermoresponsive menjanjikan. Walaupun terdapat satu faktor penting yang menjadikan PNVCL sangat menarik; ia telah dilaporkan bahawa PNVCL perlu meningkatkan ciri-ciri mekanikal, biocompatibility, dan macroporosity untuk menjadikannya calon yang menarik untuk aplikasi bioperubatan. Oleh itu, nanoteknologi telah digunakan sebagai satu cara untuk meningkatkan sifat-sifat PNVCL dengan menambah jumlah sedikit nanofiller samping memelihara ciri-ciri suhu yang responsif. Dalam kerja-kerja ini, parameter yang ketara pempolimeran telah mengenal pasti, kemudian melaksanakan mensintesis dan pencirian polimer thermoresponsive / pengisi nanocomposites. Satu siri nanocomposites PNVCL telah dibangunkan dan digabungkan dengan nanofillers (organo (C20) dan maghemite nanotube karbon multiwalled (Fe-MWCT)) melalui proses pempolimeran in-situ dibantu oleh kacau magnet. nanocomposites PNVCL yang disintesis tertakluk kepada proses pencirian yang berbeza seperti kestabilan haba, penukaran, perubahan morfologi, bengkak dan sifat-sifat reologi menggunakan kaedah pencirian yang berbeza seperti sinar-X pembelauan (XRD), Fourier Transform Infrared Spektroskopi (FTIR), Nuklear spektroskopi resonans magnet (NMR), dan Scanning Electron Microscopy (SEM). Kestabilan haba yang nanocomposites telah ditentukan dengan menggunakan analisis Termogravimetri (TGA) dan Differential Scanning Calorimetry (DSC). Daripada keputusan, yang diperhatikan rendah dan luas puncak XRD daripada nanocomposites disahkan rejim sudut yang lebih rendah daripada sampel disebabkan oleh pengembangan jarak basal. Peningkatan dalam kandungan tanah liat dan Fe-MWCNTs meningkat pengembangan d-jarak daripada nanocomposites. Selain itu, keputusan FTIR menunjukkan band penyerapan min kumpulan berfungsi utama dalam nanocomposites seperti hidrogen percuma karbonil (C = O), hidrogen terikat -OH regangan dan percuma -OH regangan. Selain itu, ^{1}H dan ^{13}C NMR telah digunakan untuk struktur polimer dan produk degradasi pencirian nanocomposites PNVCL. Keputusan TGA menunjukkan peningkatan yang ketara dalam nanocomposites PNVCL selepas penubuhan C20 dan Fe-MWCNTs. Keserasian Fe-MWCNTs dengan PNVCL didapati lebih tinggi berbanding dengan C20 dalam matriks polimer. Ini terbukti yang lebih tinggi d-jarak, haba, dan sifat-sifat mekanik Komposit nano yang dibentuk dengan 0.3% berat Fe-MWCNT berbanding Komposit nano dibentuk dengan 3% berat C20. Struktur diinterkalasi daripada nanocomposites PNVCL diberikan kestabilan haba yang baik kepada nanocomposites seperti yang ditentukan oleh TGA. Lengkung DTG menunjukkan tiada perbezaan yang signifikan kandungan C20 atau Fe-MWCNT lebih tinggi ke atas proses penyahkutuban, menunjukkan kesan positif daripada pengisi kepada proses degradasi terma. Reka bentuk pusat komposit (CCD) daripada kaedah gerak balas permukaan (RSM) telah digunakan semasa proses pengoptimuman dalam kajian ini. Proses pengoptimuman dilakukan dengan tiga faktor proses (suhu, masa, dan jumlah nanofillers) dan keputusan menunjukkan kenaikan suhu dan nanofillers kandungan untuk memihak kepada pempolimeran daripada nanocomposites ke tahap tertentu. Model kuadratik dibangunkan adalah agak tepat. Kekurangan yang tidak ketara kesilapan peratusan patut dan rendah semasa eksperimen pengesahan menunjukkan kesahihan proses pengoptimuman pada tahap yang penting bagi kedua-dua nanocomposites.

ABSTRACT

Poly (N-vinylcaprolactam) (PNVCL) has attracted much research attention recently as one of the promising thermoresponsive polymers. Even there is an essential factor that makes PNVCL very attractive; it has been reported that PNVCL needs to improve its mechanical characteristics, biocompatibility, and macroporosity to make it an exciting candidate for biomedical applications. Therefore, nanotechnology has been used as a way to enhanced PNVCL properties by adding a little amount of nanofiller while preserving the temperature-responsive properties. In this work, the significant parameters of polymerization have been identifying, then perform the synthesize and characterization of thermoresponsive polymer/fillers nanocomposites. A series of PNVCL nanocomposites were developed and incorporated with nanofillers (organoclay (C20) and maghemite multiwalled carbon nanotubes (Fe-MWCT)) via an in-situ polymerization process assisted by magnetic stirring. The synthesized PNVCL nanocomposites were subjected to different characterization processes such as thermal stability, conversion, morphology changes, swelling and rheological properties using different characterization methods like X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Nuclear magnetic resonance spectroscopy (NMR), and Scanning Electron Microscopy (SEM). The thermal stability of the nanocomposites was determined using Thermogravimetric analysis (TGA) and Differential Scanning Calorimetry (DSC). From the results, the observed low and broad XRD peaks of the nanocomposites confirmed the lower angle regime of the samples due to the expansion of the basal spacing. An increase in the clay and Fe-MWCNTs content increased the d-spacing expansion of the nanocomposites. Additionally, the FTIR results show the mean absorption bands of the main functional groups in the nanocomposites such as hydrogen free carbonyl (C=O), hydrogen-bonded -OH stretching and free -OH stretching. Moreover, ^{1}H and ^{13}C NMR was used for polymer structure and degradation products characterization of the PNVCL nanocomposites. The TGA results show a significant improvement in the PNVCL nanocomposites after the incorporation of C20 and Fe-MWCNTs. The compatibility of Fe-MWCNTs with PNVCL was found to be higher compared to that of C20 in the polymer matrix. This was evidenced in the higher d-spacing, thermal, and mechanical properties of the nanocomposite formed with 0.3 wt% Fe-MWCNT compared to nanocomposite formed with 3 wt% C20. The intercalated structure of the PNVCL nanocomposites conferred improved thermal stability to the nanocomposites as determined by TGA. The DTG curves show no significant influence of higher C20 or Fe-MWCNT contents on the depolarization process, indicating a positive effect of the fillers on the thermal degradation process. The central composite design (CCD) of the response surface methodology (RSM) was employed during the optimization process in this study. The optimization process was performed with three process factors (temperature, time, and the amount of nanofillers) and the results show increases in the temperature and nanofillers content to favor the polymerization of the nanocomposites to a certain extent. The quadratic model developed was reasonably accurate. The insignificant lack of fit and low percentage errors during the validation experiment showed the validity of the optimization processes at a significant level for both nanocomposites.

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LIST OF SYMBOLS

Å	Angstrom
d	inter-clay layer space
g	Gram
G′	Storage modulus
G″	Loss modulus
hr	Hour
I	Initiator
L	Liter
m	Metre
M	Monomer
min	Minute
^{o}	Degree
^{o}C	Degree Celsius
P	probability values
R^2	Coefficint og regression
T	Temperature
t	Time
T%	transmittance
T_g	Glass transition temperature
w%	Weight percentage
θ	Scattering angle
λ	wavelength of the X-ray

LIST OF ABBREVIATIONS

^{13}C-NMR	Carbon nuclear magnetic resonance
^{1}H-NMR	Proton nuclear magnetic resonance
5-FU	5-fluorouraci
AGA	Acrylamidoglycolic acid
AIBN	Azoisobutylnitrile
Am	Acrylamide
ATRP	Atom transfer radical polymerization
BAC	N, N′-bis(acryloyl)cystamine
$C_{11}EO_{42}$	poly(ethylene oxide)
C20	Cloisite 20
CCD	Central Composite Design
CEC	Cation exchange capacity
CH_3I	Methyl iodide
CMCS	Carboxymethyl chitosan
CNT-PCs	Carbon nanotube- Polymer Composites
CNTs	Carbon nanotubes
CPT	Camptothecin
Cr(VI)	Chromium primarily
CTAs	Chain-transfer agents
DEAEMA	2-Diethylaminoethyl methacrylate
DEX	Dextran
DHG	Double-hydrophilic glycopolymer
DMAEM	2-Dimethyl aminoethylmethacrylate
DOE	Design of experiment
DOX	Doxorubicin
DSC	Differential scanning calorimetry
DTG	derivative thermal gravimetric
EVOH	Poly(ethylene-co-vinyl alcohol)
FA	Folic acid
Fe_3O_4 -MWCNTs- COOH	Carboxilic acid functionalized multiwall carbon nanotubes and magnetic iron oxide nanoparticles

Fe_3O_4	Magnetite iron oxide
Fe-MWCNTs	Maghemite–multiwalled carbon nanotubes
fib	Fibrinogen
FITC	fluorescein isothiocyanate
FP	Frontal polymerization
FRP	Free radical polymerization
FTIR	Fourier-transform infrared spectroscopy
GC-MS	Gas Chromatography/Mass Spectroscopy
GO	Graphene oxide
HDPE	Polyethylene
HEMA	2-Hydoxyethyl methacrylate
HPCL	Poly(Ɛ-caprolactone)
HPMA	N-(2-hydroxypropyl)methacrylamide
IANa	Itaconic acid sodium
IPTES	3-isocyanato- propyltriethoxysilane
LCST	Low critical solution temperature
LDPE	low-density polyethylene
Meg	Megestrol acetate
MMA	Methyl methacrylate
MMANa	Metacrylic acid sodium
MMT	Montmorillonite
MR	Magnetic resonance
MWCNTs	Multi-walled carbon nanotubes
MWCNTs/PANI/Fe_3O_4	MWCNTs/polyaniline/magnetite
MWCNTs-AuNPs	multiwall carbon nanotubes-gold nanoparticles
MWNT-PAmI	MWNT grafted cationic polyelectrolyte
nBMA	n-butyl methacrylate
NGs	Nanogels
NH_2	Aminopropyl
NMR	Nuclear Magnetic Resonance
NMR	Nuclear magnetic resonance
ONZ	Ornidazole
P(OVNG)	6-O-vinyl-nonanedioyl-D-galactose

P4VP	Poly (4-vinyl pyridine)
PA6	Polyamide 6
PAA	Polyacrylic acid
PAA	Poly(acrylic acid)
PAA	Polyacrylamide
PAMAM-0	Polyamidoamine generation-0
PAN	Polyacrylonitrile
PBD	Plackett-Burman Design
PBMA	Poly(n-butyl methacrylate)
PCL	poly(e-caprolactone)
PDEAEMA	Poly(2-diethylaminoethyl methacrylate)
PEG	Poly(ethylene glycol)
PEG	poly(ethylene glycol)
PEG	Poly(ethylene glycol)
PEGMA	Poly(ethylene glycol)methacrylate
PEI	Polyethyleneimine
PEI	Polyethyleneimine
PEO	Poly(ethylene oxide)
PEO–Me	Poly(ethylene-oxide) dimethylether
PHPMA	Poly(N-(2-hydroxypropyl)methacrylamide)
PHPMA	Poly(N-(2- hydroxypropyl)methacrylamide)
PLA	Polylactide
PLA	Polylactide
PMMA	poly(methyl methacrylate)
PMMA	Polymethyl methacrylate
PNC	polymer nanocomposites
PNIPAM	Poly-(N-isopropylacrylamide)
PNVCL	Poly (N-vinylcaprolactam)
PNVCL–COOH	poly(N-vinylcaprolactam) -Carboxyl-terminated
POE-g-AA	Polyethylene–octene elastomer-grafted
	metallocene polyethylene–octene elastomer
PP	Polypropylene
PS	polystyrene

PSS	poly(sodium 4-styrenesulfonate)
PtBA-b-PS	Poly [(tert-butyl acrylate)-b-styrene]
PTME	Polytetramethylene ether
PU	Polyurethanes
PUU	Poly(urea urethane)
PVC	Polyvinyl chloride
PVC	poly(vinyl chloride)
PVC	polyvinyl chloride
PVK	Poly(N-vinyl carbazole)
PVOH	Poly(vinyl alcohol)
PVP	poly (4-vinylpyridine)
RAFTP	Reversible addition fragmentation chain transfer polymerization
RhB	Rhodamine B
ROP	Ring opening polymerization
RSM	Response surface methodology
R-Sq	R-squared
R-Sq(adj)	adjusted R-squared
R-sq(pred)	Predicted R-squared
S	styrene
SA	Sodium alginate
SEM	Scanning electron microscopy
SI-ATRP	surface-initiated atom transfer radical polymerization
SR	Swelling ratio
SWCNTs	Single walled Carbon Nanotubes
TEM	Transmission electron microscopy
TGA	Thermogravimetric analysis
THF	Tetrahydrofuran
UA	Undecenoic acid
UCST	Upper critical solution temperature
UV-Vis	Ultraviolet-visible
VCL	vinylcaprolactam

VPTT	Volume phase transition temperature
WPU	Waterborne polyurethane
XRD	X-ray diffraction

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